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WATER POLLUTION CONTROL DEMONSTRATION

Villanova University
Villanova, Pennsylvania

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FINAL PROGRESS REPORT

ON

WATER POLLUTION CONTROL DEMONSTRATION

PROJECT GRANT NO. WPD-174-01-67

Submitted to

FEDERAL WATER POLLUTION CONTROL ADMINISTRATION

by

VILLANOVA UNIVERSITY

Villanova, Pennsylvania

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Attention:

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Federal Water Pollution Control Administration

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WATER POLLUTION CONTROL DEMONSTRATION PROJECT

GRANT NO. WPD-174-01-67

Identification of Grantee and Project

Grant No. WPD-174-01-67 has been made to Villanova University for the purpose of demonstrating a process for elimination of water pollution by waste oil re-refining plants.

Summary of Work Accomplished

Laboratory Studies

The initial work on the project consisted in carrying out each step of the process in small bench scale equipment at Villanova University. Physical inspections on some products were checked by the laboratories of Atlantic - Richfield Company and the Gulf Oil Corporation. The next step was to carry out the various steps on commercial size equipment.

Plant Operation

It so happened that one of the two waste oil re-refining plants of Berks Associates, Inc., located at Douglasville, Pa. contained the necessary large commercial size equipment needed to demonstrate the feasibility of re-refining waste oil economically without water pollution. This plant had been operated for the last 5 years without using sulfuric acid, but the operation was not entirely satisfactory. Some small amount of water pollutants were produced and the process had been operating at a financial loss for over a year.

Because of the knowledge that was gained in the laboratory studies on the bench scale equipment, it was possible to make some small mechanical changes on the Douglasville plant. The results were gratifying. The water pollution by this plant was eliminated, some quality improvement was obtained, and the process became economically profitable.

The actual daily production by this plant of over 20,000 gallons per day of good quality lubricating oils with profitable operation and clean water effluent is the best possible demonstration of process feasibility.

Description of New Process

To recover high quality fuel oils and lubricating oils from waste oils, it is necessary to remove five classes of contamination products. These contaminants are:

(1) water
(2) solids

(3) low and intermediate boiling materials
(4) resins and decomposition products of oil, fuel, and additives
(5) color bodies

There are at least several methods and a variety of types of equipment that might be employed for carrying out each of these steps, as well as a number of possible combinations of steps.

The process investigated in the laboratory is as follows:

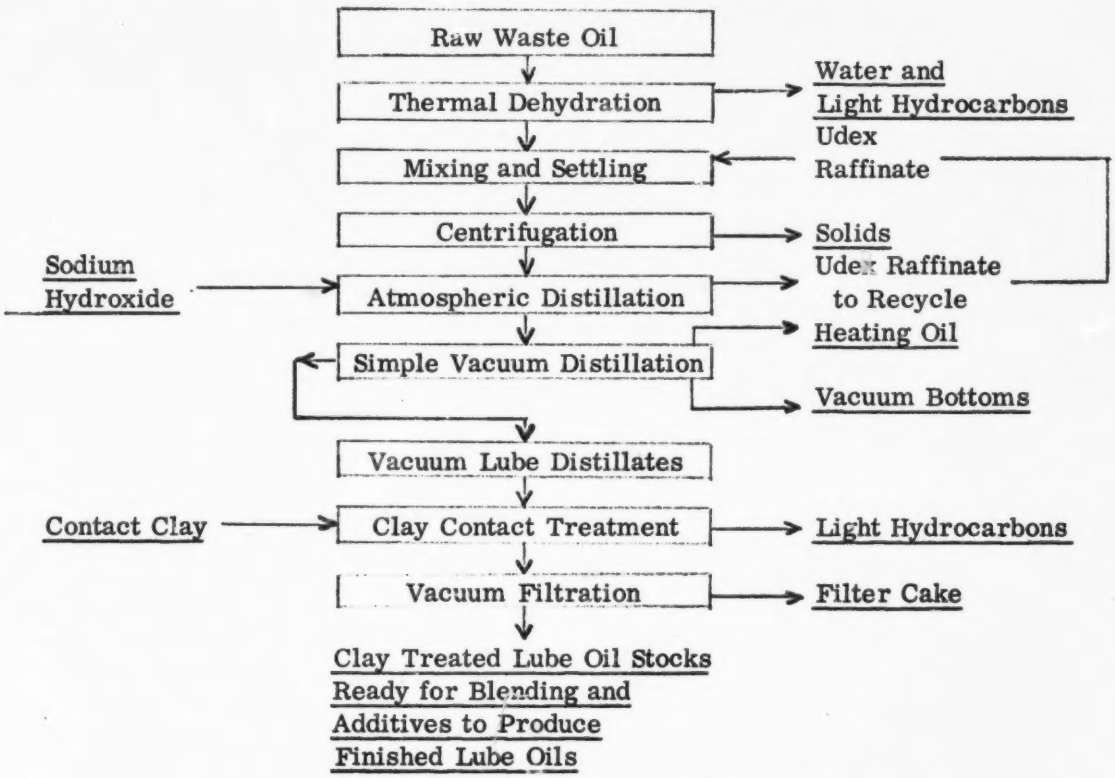
(1) the waste oil is heated to remove water and low boiling materials. A temperature of 400°F. at atmospheric pressure was found to be adequate for reduction of water content to below 0.1%.
(2) the dehydrated oil is diluted with a light naphtha to agglomerate or coagulate the suspended solids and aid in their removal. It was found that an equal volume of Udex Raffinate (mainly normal heptane) was a satisfactory diluent. The detailed procedure for removal of suspended solids from the dehydrated oil is as follows: The dehydrated oil is mixed for at least 10 minutes with an equal volume of Udex Raffinate. The Udex Raffinate is a by-product of aromatic hydrocarbons manufacture. It consists of paraffinic hydrocarbons having an approximate boiling range from 155°F. to 200°F. The solution stands for 24 hours. During this time the very fine suspended solids flocculate and are then easily removed by a disk type of continuous centrifuge. Based on small scale centrifugations carried out in the laboratory, three centrifuge manufacturers (Centrico-Westphalia, Sharples and De Laval) are prepared to quote prices to a potential customer for their various types of commercial size centrifuges. For their various size machines they will list throughput rates at continuous operation with the removal of visually observable suspended solids from a 1:1 by volume solution of dehydrated waste oils and Udex Raffinate.
(3) removal of the solvent naphtha (Udex Raffinate). The Udex Raffinate and accompanying traces of light materials are distilled overhead by heating the oil at atmospheric pressure until a vapor temperature of 400°F. is reached. One weight percent of dry sodium hydroxide is added at the start of the distillation to neutralize acidic materials and prevent deposition of sticky resinous materials in the condenser.
(4) vacuum distillation of the oil. The oil is vacuum distilled to minimum bottoms (at 28 to 29 inches of mercury vacuum without cracking) in order to obtain vacuum distillate fractions. The lightest of these fractions are fuel oils and the highest boiling fractions are lubricating distillates.
(5) the lubricating distillates are clay treated to prepare finished lubricating oils. To produce a No. 3 color oil, about 4 weight percent of a good quality filtering clay is heated with the oil to 640°F. in an inert atmosphere during a 60 minute period. After cooling to 300°F., the spent clay is removed by vacuum filtration.

(6) the vacuum bottoms contains solid metallic compounds (probably as organometallics) that render it unsuitable as a residual fuel. It may be diluted with three volumes of Udex Raffinate and solids then can easily be filtered off. After solvent removal, the filtrate is a satisfactory residual fuel.

(7) light hydrocarbons are recovered:
   (1) during the dehydration step
   (2) as a pre-lube distillate during the vacuum fractionation step
   These materials are both satisfactory as a fuel for the process.

(8) the only by-products of the above process are water and solids. In a commercial operation, both are easily disposed of without producing water pollution.

The following block diagram presents the new process graphically:



End Uses for Waste Oils

The conventional uses for re-refined oils are as follows:

(a) as re-refined lubricants for internal combustion engines
(b) as industrial lubricants
(c) as farm equipment lubricants
(d) as railroad journal box lubricants
(e) as a component of industrial fuel oils

The origin of the waste oils obtained by collectors and delivered to re-refining plants is about 80 to 85 percent crankcase drainings and 15 to 20 percent non-lube oils. The crankcase drainings are predominantly from autos, gasoline burning trucks, and diesel fuel burning trucks. The non-lube oils are chiefly industrial waste oils from such diverse sources as steel mills, metal processing plants, and vegetable oil processing plants.

The waste oil reclaiming plants can reprocess waste lube oils into re-refined lube oils, but the non-lube oils such as cutting oils, soluble oils, or vegetable oils cannot be converted into automotive lubricating oils. Except for a variety of relatively minor uses, reclaimed non-lube waste oils now end up as a component of heavy industrial fuel oils.

Potential uses for by-products

The re-refining process that was investigated in the laboratory produced two by-products. They are:

(1) the solids removed by centrifugation
(2) the vacuum tower bottoms. This material may be diluted with Udex Raffinate and solids then can be filtered off easily. After removal of the solvent, the filtrate is a liquid.

No organized study was made of either the solid or the liquid by-product. A sketchy investigation was made as shown in the Appendix, page 50. This preliminary work suggested, however, that the solids might make excellent carbon black or adsorbent char and that the liquid from the vacuum bottom might make a rubber extender.

A reliable source is needed for both of these materials from full plant scale production before a further investigation of these materials would be justified or the results would be meaningful. However, if such a study were to be successfully carried out in the future, the economic value of each of the two by-products could considerably add to the profitability of the waste oil re-refining process.

Another potential application for the solid by-product is as a cut-back asphaltic material for coating gravel and sand in bituminous road construction.

The semi-solid residue that settles from waste oil during storage has been used commercially for over 5 years in Canada as a road oil and as a bituminous binder for fine and coarse aggregates.

Further investigation of this application for the solid by-product also requires a reliable source of starting material from full scale plant production.

Detailed Description of Work Done

Initial Work

The bench scale investigation began with a study of the water content and the solids content of waste oils from a variety of sources. Samples of waste oils were obtained as auto and truck crankcase drainings from service stations, from collectors, from re-refining plants, and as industrial waste oils from the U. S. Steel Company, Results are shown in Table No. 1. The water content of the raw waste oils was measured by the Water by Distillation Test ASTM-D-95-58. The solids content was measured by the Coagulated Pentane Insolubles Test ASTM-D-893-60T. It is difficult to reproduce the results of the Coagulated Pentane Insolubles Test on raw wet samples of waste oils. Hence this data is an approximation only.

No two samples of waste oil from different sources were found to be the same, and samples obtained at different times from the same source also varied. One of the most difficult waste oils to re-refine in the laboratory was obtained from the Mohawk Re-refining Co. of Newark, New Jersey. This waste oil contained considerable water, dirt, and tarry or sticky materials. It was felt that if this waste oil could be converted into good quality lubricating oil in the laboratory, the process most probably would be quite suitable for any waste oil that might be encountered. Therefore, the major effort in the laboratory was based on the study of Mohawk waste oil.

Bench Scale Studies

(a) conversion of waste oils into lubricating oils

Two samples of raw waste oil were withdrawn from a 50 gallon drum, after thorough agitation of the drum. The drum was received from the Mohawk Oils Refining Company on June 1, 1967. These samples were then separately dehydrated to a temperature of 400°F. using a small quantity of steam. The dehydrated samples were then combined. Physical inspections of the raw oil and of the combined dehydrated oil are given in Table No. 2.

The dehydrated oil was then mixed with an equal volume of Udex Raffinate and stirred for 5 minutes at 150°F. The material was settled for 24 hours at room temperature and centrifuged in a continuous Centrico-Westphalia laboratory centrifuge at 3000 G. to remove solids.

The centrifuged oil was stripped of light solvent to 400°F. with steam. The physical properties of the stripped oil are given in Table No. 2.

A 3,300 ml. sample of the stripped oil was charged to a 5 liter flask fitted with an overhead water-cooled condenser and a vacuum receiver that was connected to a mercury manometer and a vacuum pump. A simple batch type of vacuum distillation was carried out. A vacuum of 29.5 inches of mercury was maintained until very slight cracking occurred near the end of the distillation, when the vacuum was slightly decreased. The distillation was ended when the vaporization rate substantially decreased. The overhead fractions collected are shown in Table No. 3. The bottoms material stuck to the inside of the flask and was not weighed. The fraction collected as overhead constituted 96.5 weight percent of the charge.

Physical inspections of the overhead fractions are shown in Table No. 4. The overhead fractions, cuts 1 to 12 inclusive, were combined into four samples and clay treated as shown in Table No. 5.

Oil samples having a Saybolt viscosity below 80 SUS at 100°F. are ordinarily not considered to be lubricating oils. Therefore, the lightest fraction of Table No. 5 (combined cuts 1,2,3) is seen to be a pre-lube fraction. This cut would normally be sold as a heating oil without clay treatment. Likewise, cut No. 13 of Table No. 4 would normally become a component of plant fuel.

There is very close agreement between the physical inspections of the three lube fractions shown in Table No. 5 and the typical manufacturing specifications used for plant control of clay treated lube oil stocks at Berks Associates, Inc. A comparison is as follows:

	Physical Properties:	
	Combined Cuts 4,5,6 of Table 5	Typical Daily No.2 Cut Manufacturing Properties
Viscosity:		
SUS at 100°F.	116.3	100 to 200
SUS at 210°F.	40.27	40 to 47
Viscosity Index	82	80 to 100
Color, NPA Number	2.0	2.5 to 3.5

	Combined Cuts 7, 8, 9 of Table 5	Combined Cuts 10, 11, 12 of Table 5	Typical Daily No. 3 Cut Manufacturing Properties
Viscosity:			
SUS at 100°F.	227.2	460.4	200 to 500
SUS at 210°F.	46.98	59.01	48 to 58
Viscosity Index	82	85	80 to 100
Color, NPA Number	3.5	7.0	3.0 to 6.0

The typical manufacturing specifications are representative of finished lube oil stocks such as are now being commercially marketed on a daily basis by Berks Associates, Inc.(1) Only blending to produce specification grade viscosities and the addition of normal amounts of conventional additives are required in order to produce salable finished lube oils from these clay treated lube oil stocks.

Based on the data of Tables 3, 4, and 5, the following yields of salable products are estimated from treating the raw waste oil:

	Volume Percent
Raw Waste Oil	100.0
Loss on Dehydration(2)	16.1
Loss on Centrifugation and Solvent Stripping(3)	10.0
Distillation Bottoms(4)	2.6
Loss on Clay Treatment(3)	5.5
Salable Products	
Lube Stocks for Blending(5)	51.6
Heating Oil(6)	14.2

Note: (1) manufacturing specifications for clay treated lube oil stocks are given in the Appendix, Page 34.
(2) calculated from measured weight loss.
(3) estimated, as quantitative data not available.
(4) calculated from weight data in Table 3.
(5) calculated as combined cuts 4 through 12 inclusive of Table 5.
(6) calculated as combined cuts 1, 2, 3, and 13 of Table 4. The combination of high VI and high VGC of cut 13 of Table 4 indicated that it contained both additives and cracked products, and hence was not suitable for clay treatment to produce a lube oil having good color.

In addition to the above salable products, there are some liquid hydrocarbons that are recovered along with aqueous condensate from the dehydration. These hydrocarbons are suitable for use as plant fuel.

The distillation carried out as shown in Tables 3, 4, and 5 was made without chemical pretreatment of the feed. A similar distillation was now carried out in the same equipment using another sample of the same stripped oil with physical properties shown in Table No. 2. The results of this second distillation are shown in Tables 6, 7, and 8.

Although the charge stock was the same for the second distillation as for the first distillation, they differed in that before the second distillation was begun, 1.0 weight percent of solid sodium hydroxide was added to the charge in the distillation flask. Also, the first distillation was carried to a liquid temperature of 810°F. and the second was carried to a liquid temperature of only 698°F.

When the second distillation was complete, all the solid sodium hydroxide had become soluble in the residual oil and the bottoms in the flash was a viscous liquid with no evidence of any solid sodium hydroxide present.

It is assumed that the sodium hydroxide neutralized the acidic materials in the oil, and rendered them non-volatile as the condenser was free of gummy deposits which appeared in the first distillation and darkened the color of the heavier distillate fractions as shown in Table No. 4.

The second distillation charge to the 5 liter flask was 3500 ml. of the stripped oil. The entire distillation was carried out as nearly as possible like the previous distillation except for the lower final liquid temperature.

Physical inspections of the overhead fractions are shown in Table No. 7. The overhead fractions, cuts 4 to 12 inclusive, were combined into four samples and clay treated as shown in Table No. 8.

Based on the data of Tables 6, 7, and 8, the following yields of salable products are estimated from treating the raw waste oil:

	Volume Percent
Raw Waste Oil	100.0
Loss on Dehydration(1)	16.1
Loss on Centrifugation and Solvent Stripping(2)	10.0
Distillation Bottoms(3) to be used for naphtha dilution and suspended solids removal to make plant fuel	9.6
Loss on Clay Treatment(2)	5.5
Salable Products:	
Lube Stocks for Blending(4)	44.0
Heating Oil(5)	14.8

In addition, some liquid hydrocarbons recovered from the dehydration are suitable for use as plant fuel.

The physical inspections shown in Table No. 8 indicate that when 4 weight percent of the Pembina Clay R-105 was used for the clay treating, samples fell within the specifications for finished lube oil stocks. Only 2 weight percent of the low cost fuller's earth was required for the combined cuts 4, 5, 6 and for 7, 8. Cuts 1, 2, 3 would normally be sold as heating oil without clay treatment.

Comparing Tables 5 and 8, one sees that the 59 SUV oil at 210°F. shown in Table 5 (distillation without use of sodium hydroxide) had a 7 NPA color while the 59 SUV oil shown in Table 8 (distillation with 1 percent sodium hydroxide) had a No. 2 NPA color. The same Pembina Clay R-105 was used for the clay treating in both cases at a 4 weight percent rate.

A third distillation was carried out in the same equipment as the previous two distillations. Another sample of the same stripped oil was used. Due to standing for several months, the sample had precipitated a small quantity of tarry material, and the specific gravity of the sample was 0.887 at 60°F./60°F. The coagulated pentane insolubles was 0.61 weight percent. The results of this third distillation are shown in Tables 9, 10, and 11. The charge to the 5 liter flask was 3,500 ml. of the stripped oil. The entire distillation was run as nearly as possible like the second distillation except that the final liquid temperature was 752°F.

Note: (1) calculated from measured weight loss
(2) estimated, as quantitative data not available
(3) calculated from weight data in Table 6.
(4) calculated as combined cuts 4 through 12 inclusive of Table 8.
(5) calculated as combined cuts 1, 2, 3 of Table 7.

Physical inspections of the overhead fractions are shown in Table No. 10. The overhead fractions, cuts 3 to 9 inclusive, were combined into three samples and clay treated as shown in Table No. 11.

Based on the data of Tables 9, 10, and 11, the following yields of salable products are estimated from treating the raw waste oil:

	Volume Percent
Raw Waste Oil	100.0
Loss on Dehydration(1)	16.1
Loss on Centrifugation and Solvent Stripping(2)	10.0
Distillation Bottoms(3)	
For naphtha dilution and suspended solids removal to make plant fuel	9.6
Loss on Clay Treatment(2)	5.5
Salable Products:	
Lube Stocks for Blending(4)	50.7
Heating Oil(5)	8.1

In addition, some liquid hydrocarbons recovered from the dehydration are suitable for use as plant fuel.

Comparing the physical inspections shown in Table 5, 8, and 11, one sees that when solid NaOH is added to the distillation charge, there is a decided reduction in the color of the clay treated lube stocks as well as an improvement in viscosity index.

Note: (1) calculated from measured weight loss
(2) estimated, as quantitative data not available
(3) calculated from weight data in Table 9.
(4) calculated as combined cuts 3 through 9 inclusive of Table 11.
(5) calculated as combined cuts 1 and 2 of Table 10.

(b) conversion of waste oils into heating oils

Waste oils that are obtained from crankcase drainings can be re-refined into lubricating oils. However, waste oils are also obtained from sources other than crankcase drainings such as machine oils, soluble oils, vegetable oils, slop fuel oils, tank bottom cleanings, rendering plant wastes etc. Such waste oils cannot economically be processed into motor oil lubricants. Efforts have been made to dispose of them in petroleum refineries by blending them into heavy residual fuels or into catalytic cracker charge stocks. These methods of disposal have proven undesirable as the solids and additives in the waste oils had objectionable effects on residual oil burners and on catalytic cracker catalyst life.

It would be desirable to be able to process all types of waste oils into No. 4 heating oils, or for selectively segregated materials, into No. 4 stationary diesel fuel after blending with purchased cutter stock.

The process investigated in the laboratory to upgrade waste oils into No. 4 heating oils is as follows:

(1) the waste oil is heated to 350°F. to remove water and sufficient low boiling hydrocarbons to insure a flash of 160°F. or higher for the dehydrated oil before the addition of cutter stock.

(2) the dehydrated oil is diluted with a light naphtha, mixed, and centrifuged to remove suspended solids. The detailed procedure for this operation is the same as the procedure described on page 2 of this report for removal of suspended solids from the waste oil being re-refined to lube oil stock. The only difference is that in this laboratory work, 3 volumes of Udex Raffinate were used per volume of dehydrated oil in order to precipitate the major portion of the compounds responsible for sulfated ash, sulfur, and carbon residue. Only one hour settling was provided before centrifugation. A clear solution was obtained and a solid cake that was about 50 percent solids.

(3) the solution is heated to 400°F. to evaporate the Udex Raffinate.

(4) physical tests are obtained on the oil sample to determine the amount of light cutter stock that is needed to bring the oil within the viscosity limit for a specification No. 4 heating oil.

Small scale laboratory tests were carried out on samples of six different waste oils. The results are shown in Tables 12-15.

The samples shown in Table No. 12 vary from two dirty waste oils from re-refiners (Sample 10 and 11), a less dirty waste oil from a re-refiner (Sample 9), a waste oil loaded with dirt from a truck service station (Sample 7), a relatively clean sample supposed to be pure crankcase drainings (Sample 6), and a relatively dirty sample supposed to be pure crankcase drainings (Sample 8).

A CRC filter was used to try to remove suspended solids from the solvent diluted dehydrated oils. The data of Table 13 shows a failure to remove the suspended solids by the CRC Filter. This failure resulted both when the CRC Filter was used with a filtering clay precoat (Sample 11), and when a filtering carbon was used (Sample 10). By contrast, the Sharples centrifuge reduced the coagulated pentane insolubles to 0.07 weight percent for Sample 11 (0.13 weight percent for duplicate run). The centrifuge also gave a clear solution for all six oil samples.

Table 14 shows physical inspections of the centrifuged samples after the solvent was removed by heating to 400°F. Table 15 shows the cutter stock required to bring the samples of Table 14 to the viscosity specifications for No. 4 heating oils.

Inspection of Table 14 shows remarkably high values of viscosity index (123 to 129) for all six samples. These high viscosity index values (along with the very low values of pour, and the high values of sulfated residue and carbon residue) suggest that considerable amounts of additives of the detergent - V.I. improver type are still present in all of the oil samples.

Plant scale experience at Berks Associates Re-refining Plant has shown that the addition of a fraction of a percent of sodium hydroxide to the hot dehydrated oil causes considerable settling out of the types of materials that cause positive values of sulfated residue and carbon residue. Hence, the sulfated residue and the carbon residue could be lowered by adding sodium hydroxide during the dehydration in step (1) of the laboratory procedure. A reduction in sulfated residue would be necessary for the samples to meet all the specifications for No. 4 heating oils.

(c) specifications for No. 4 heating oils

A No. 4 heating oil is used frequently in an oil burner installation not equipped with preheating facilities. It is defined by ASTM-D-396-66 specifications as follows:

Flash Point, °F. minimum (O.C.)	130
Pour Point, °F. maximum	20
Water and Sediment, % by Vol. maximum	0.50
Ash, Wt. % maximum	0.10
Viscosity, SUV/100°F.:	
minimum	45
maximum	125
Kinematic Viscosity, centistokes:	
minimum	5.8
maximum	26.4
Sulfur, Wt. % maximum	1.0

The manufacturing specifications of a major oil company selling a large volume of No. 4 heating oil on the east coast are as follows:

Flash Point, °F. minimum (O.C.)	150
Viscosity, SUV/100°F.:	
minimum	60
maximum	100
Kinematic Viscosity, centistokes:	
minimum	10.3
maximum	20.5
Pour Point, °F. maximum	0
Water and Sediment, % by Vol. maximum	0.5
Sulfur Content, Wt. % by ASTM-D-129	
maximum	1.8
Ash, Wt. % maximum	0.10

The specifications listed above for No. 4 heating oil are fully complied with, if the sulfated residue is reduced by addition of sodium hydroxide during the dehydration step of the laboratory procedure. This may be seen by comparing the specifications listed above with the data of Tables 14 and 15.

In order to establish that a material may be marketed as a No. 4 heating oil, in addition to meeting the physical specifications, it must also perform satisfactorily in large scale burning tests. These tests require a number of barrels of the heating oil. For such testing to have meaningful results, a uniform and reliable source of the heating oil is needed. This can be obtained only from full plant scale production. Berks Associates at Douglasville, Penna. possess the necessary facilities for carrying out this work and have agreed to prepare heating oil stocks and provide for the necessary burning tests in the future. If the economics are favorable, the process will be demonstrated by operation on a continuous plant scale by Berks Associates, as is now being done for production of lubricating oils from waste oils.

(d) conversion of waste oil into diesel fuel

In many parts of the United States the sales value of No. 4-D diesel fuel (under present market conditions 8.5 to 9.0 cents a gallon) is about a cent a gallon higher than the sales value of No. 4 heating oil. The maximum value of sulfated residue is more restrictive for No. 4-D diesel fuel than for No. 4 heating oil. The method of producing No. 4-D diesel fuel from waste oils would be the same as the method of producing No. 4 heating oil from waste oils, except that somewhat more sodium hydroxide would have to be added to the hot oil during the dehydration.

To satisfactorily market a No. 4 diesel fuel, it should be approved by both the Diesel Engineering Manufacturers Association and the Office of the Vice President of Research of the Association of American Railroads. After an actual satisfactory performance test of a diesel fuel by these agencies, it would be approved for medium and low speed diesel engines. Such a test will require a number of

barrels of diesel fuel.

(e) specifications for diesel fuels

Four diesel fuels are marketed in substantial quantities on the eastern seaboard. They are:

No. 1-D diesel fuel — Used by city buses and similar vehicles making many stops, where smoking of the burned fuel is highly undesirable.

No. 2-D diesel fuel — Used by over the road diesel trucks making long hauls.

No. 3-D diesel fuel — A blend of No. 1-D and No. 2-D for intermediate service.

No. 4-D diesel fuel — A heavier blend used by caterpillar tractors, stationary power plant diesel engines, and railroad diesel engines.

Prior to a full scale operating test to measure fuel performance, the preliminary specifications that a No. 4-D diesel fuel should meet are listed under tests ASTM-D-396-66 and ASTM-D-975-66T.

These specifications are substantially as follows:

Flash Point, O.C., °F. minimum	150
Water and Sediment, % by Vol. maximum	0.6
Viscosity, SUS/100°F. maximum	500
Carbon Residue, Wt. % maximum	3.0
Ash, Wt. % maximum	0.04
Pour Point, °F. maximum	35

A cetane index of 50 minimum is desired.

The petroleum industry has traditionally made very good quality No. 4-D diesel fuel in times of emerge cy shortages by blending off-specification lube oils with cutter stocks. It is likely that at a future time, Berks Associates can demonstrate the profitable production of satisfactory No. 4-D diesel fuel from non-lube waste oils.

Plant Scale Studies

The plant process projected from our laboratory studies and the plant process now being demonstrated by the daily commercial operation at Berks Associates, Inc. at Douglasville, Penna. are alike in their major processing steps. These steps are:

(1) waste oil dehydration and chemical treatment of the hot dehydrated oil
(2) light naphtha dilution and solids removal

(3) atmospheric distillation to recover the light naphtha for recycle
(4) vacuum fractionation to obtain high quality lube distillates and No. 1 fuel oil
(5) clay treatment of distillate lube oil stocks

Step (1) waste oil dehydration

Laboratory dehydration of many samples, with or without steam, showed that most of the water is removed between liquid temperatures of 220°F. and 270°F. Experimental commercial dehydrations at 280°F. were carried out at Berks Associates and showed in every case that the water was reduced to below 0.1 percent as measured by ASTM Test D-95-58. By carrying out the laboratory dehydrations to a temperature of 400°F., the light ends are removed which boil in the same range as the light naphtha used for dilution.

Solid sodium hydroxide is added to the dehydrated oil in the laboratory procedure in order to neutralize the acidic components of the waste oil. Proprietary materials are added to the hot dehydrated oil for this purpose at Berks Associates.

Step (2) naphtha dilution and solids removal

In the laboratory procedure, an equal volume of Udex Raffinate is mixed with the dehydrated oil. After standing to permit flocculation of solids, the solution is centrifuged. At Berks Associates, a light process by-product is used for dilution of the dehydrated oil in order to effect solids removal by an accelerated settling technique. For complete information on accelerated settling devices related to any particular plant situation, application may be made to Pielkenrood-Vinitex, Assendelft, Holland. This firm provides service internationally in this field.

Step (3) atmospheric distillation

In the laboratory procedure, simple atmospheric distillation is carried out to a temperature of 400°F. This removes light hydrocarbons equivalent in weight to the weight of the Udex Raffinate added in Step (2). At Berks Associates, a fractionating tower is used.

Step (4) vacuum fractionation

In the laboratory procedure, a simple batch type of vacuum distillation is carried out. A vacuum of 29.5 inches of mercury is maintained. An effort is made to obtain minimum bottoms without appreciable cracking. At Berks Associates, an effective vacuum fractionation is carried out at a tower top pressure of 26.8 inches of mercury vacuum. Due to the use of an effective fractionation tower, the percent tower bottoms is considerably smaller than in the simple distillation performed in the laboratory.

Step (5) clay contacting treatment

In the laboratory procedure, the oil is heated with a weighed quantity of clay to 640°F. over a 60 minute period with constant stirring in N_2 atmosphere. It is cooled to 300°F. and vacuum filtered. The plant procedure is essentially similar except that mechanical agitation is used and a rotating vacuum filter is employed.

The entire plant operation has been aptly described by Mr. Roy H. Schurr, President and owner of Berks Associates, Inc. in letter to Captain Henry T. Koonce, dated April 26, 1968, and presented in the Appendix. Proprietary equipment functions are covered by U.S. Patent No. 3,173,859 to John M. Chambers, dated March 16, 1965.

Financial Study

A detailed financial study of the costs of each re-refining operation has been made possible because of the kindness in supplying extensive and detailed plant operating cost data by Mr. Roy H. Schurr, President of Berks Associates, Inc. and Mr. L. S. Schofield, President of Schofield and deVries, Limited. Special thanks are due to Mr. R. F. Thomas, Plant Manager of Schofield and deVries, Limited, for much detailed and exhaustive work that made possible a reliable financial comparison of present and projected operations.

This work is not shown in detail since in this type of cost study the economic calculations are subject to constantly changing materials and processing costs, and after a short interval, the details of such work are no longer meaningful. However, the conclusions are valid for a considerable period. They are as follows:

(1) re-refining of waste oils using sulfuric acid cannot be profitable based on present costs of securing waste oils for processing, and at present sales values of re-refined oils.

(2) re-refining of waste oils by the demonstrated process now is and can continue to be a profitable process, and this process also eliminates water and air pollution.

(3) based on present materials costs and product sales values, the break-even size for a plant operating as outlined in this report is about 50,000 gallons per stream day of waste oil charged.

Smaller size plants can be profitable only if they market specialties such as industrial greases and specialty lubes rather than simply re-refined oils.

(4) Installation of a plant having a design capacity of 75,000 gallons per stream day should provide a 3 year payout before taxes.

Future Publications

After Berks Associates have completed the rebuilding of their entire operating complex, it is the intention of Mr. Roy H. Schurr and Mr. H. Lester Schurr to publish a learned paper on refining of waste oils. This paper will be published in a technical journal, and reprints of this paper will be made freely available.

Special thanks are due for the consultation and excellent plant operational work of Mr. H. Lester Schurr and the good efforts of Mr. Herbert A. Hadley, Mr. Malcolm L. Balmer and Mr. George W. Miller of Berks Associates. Their work resulted in a profitable plant operation that is converting obnoxious waste oils into useful products without water pollution.

TABLE 1

Physical Inspections on Raw Waste Oils

Source of Oil	Water by Distillation, Vol. % (1)	Solids by Coagulated Pentane Insolubles Wt. % (2)
Service Station Samples		
American Oil Co. Service Station	Nil	4.00
Gulf Service Station	Nil	5.11
Atlantic Service Station	0.24	6.87
Sunoco Service Station	Nil	3.41
Tidewater Flying A Service Station	Nil	7.17
Villanova Citgo Truck Service Station	0.12	7.30
Samples from Collector's Truck		
Waste Oil Collector's Sample delivered to Regal Petroleum Co. Bridgeport, N.J. 12/14/66	0.6	1.55
Typical Charge Stocks to Re-refining Plants		
Regal Petroleum Co., Bridgeport, N.J. 5/19/67	2.2	7.03
Berks Associates, Douglasville, Pa.	Nil	6.40
Mohawk Oils Refining Co., Newark, N.J.	11.0	3.30
Industrial Waste Oils		
U.S. Steel Corp., Fairless Works Claimed to be representative of 200,000 gallons per month waste oils	70.0	Above 3.00

Note: (1) ASTM Test D-95-58
(2) ASTM Test D-893-60T

TABLE 2

Waste Oil from Mohawk Oils Refining Company

(Lab Identification - Sample No. 11-rec. 6/1/67)

Raw Waste Oil from Drum

Water by Distillation, Vol. %	10.5
Specific Gravity 60°F/60°F	0.901

Dehydrated to 400°F. with Steam

Weight Loss on Dehydration, Wt. %	16.2
Water by Distillation, Vol. %	0.0
Flash, O.C., °F.	410
Fire, O.C., °F.	430

Centrifuged in Udex Solution(1) and Stripped to 400°F. with Steam

Viscosity at 210°F., SUS	55.76
Specific Gravity 60°F/60°F.	0.889
Sulfated Residue, Wt. %	1.71
Coagulated Pentane Insolubles, Wt. %	2.4

Note: (1) the dehydrated oil was mixed with an equal volume of Udex Raffinate and stirred for 5 minutes at 150°F. The material was settled for 24 hours at room temperature and centrifuged in a continuous Centrico-Westphalia laboratory centrifuge at 3000 G.

TABLE 3

Laboratory Distillation of Mohawk Waste Oil Treated as Shown in Table 1

(Lab Identification No. C-185)

Volume of Charge in Milliliters:	
Observed	3300 ml.
Calc. from wt. data	3304 ml.
Wt. of Charge in Grams	2937 grams
Physical Inspections of Charge:	
Sp. Gr. 60°F./60°F.	0.889
Sulfated Residue, Wt.%	1.71
Coag. Pentane Insol., Wt.%	2.4
Wt.% Solid NaOH Added to Charge	0.0

Cut No.	Pressure in inches of Hg. Vac.	Observed Liquid Temp. °F.	Observed[1] Vol. of Cut in Ml.	Measured Wt. of Cut in Grams.	Cumulative Percent	
					Calc. from Wt. Data Vol.%[2]	Measured Wt.%
1	Atm.	218-425	29	25.0	0.9	0.9
2	29.5	405-490	250	244.0	9.4	9.2
3	29.5	490-504	250	218.7	17.0	16.6
4	29.5	504-520	250	236.4	25.3	24.6
5	29.5	520-540	250	251.3	34.1	33.2
6	29.5	540-554	250	229.6	42.1	41.0
7	29.5	554-570	250	248.8	50.7	49.5
8	29.5	570-580	250	241.8	59.1	57.7
9	29.5	580-610	250	249.7	67.7	66.2
10	29.4	610-640	250	260.1	76.7	75.1
11	29.8	640-770	250	258.9	85.6	83.9
12	28.0	770-800	250	259.4	94.4	92.7
13	28.0	800-810	90	110.9	98.2	96.5
Bottoms + Loss		---			1.8	3.5

Note: (1) volumetric data are less accurate than weight data as it is difficult to withdraw sample from vacuum receiver without collecting additional sample after recording volume.

(2) calculated from weight data using gravities shown in Table 3.

TABLE 4

Physical Inspections of Vac. Distillation Cuts from Table 3

(Lab Identification No. C-185)

Cut No.	Specific Gravity 60°/60°F.	Viscosity at 100°F. SUS	Viscosity at 210°F. SUS	Viscosity Index	Viscosity Gravity Constant	Color NPA Number	Sulfated Residue Wt. %	Coag. Pent. Insol. Wt. %
1	- -	- -	- -	- -	- -		- -	Nil
2	0.866	37.69	22.17	- -	- -	3.5		Nil
3	0.868	87.07	37.54		0.826	3.0		Nil
4	0.866	104.2	39.91		0.819	3.0		Nil
5	0.867	131.7	39.95		0.816	3.0		Nil
6	0.870	140.7	42.66	83	0.818	3.0		Nil
7	0.873	178.8	44.27	83	0.818	4.0		Nil
8	0.875	225.7	46.72	79	0.816	3.5		Nil
9	0.877	235.4	48.62	98	0.818	8.0+		Nil
10	0.879	256.3	50.40	103	0.819	8.0	Nil	Nil
11	0.877	265.9	50.73	101	0.816	8.0+	Nil	Nil
12	0.879	234.4	48.59	99	0.821	8.0+	Nil	Nil
13	0.889	181.1	46.30	114	0.838	8.0+	Nil	Nil

TABLE 5

Physical Inspections of Clay Treated Vac. Distillation Cuts from Table

(Lab Identification No. C-185)

Cut No. (from Table 3)	Specific Gravity $60^{o}/60^{o}F.$	Viscosity at $100^{o}F$ SUS	Viscosity at $210^{o}F.$ SUS	Viscosity Index	Viscosity Gravity Constant	Color NPA Number	Sulfated Residue Wt. %	Coag. Pent. Insol. Wt. %
Combined Cuts[1] 1,2,3	0.868	76.71	36.83		0.829	1.5	0.003	Nil.
Combined Cuts[1] 4,5,6	0.867	116.3	40.27	82	0.818	2.0	Nil.	Nil.
Combined Cuts[1] 7,8,9	0.874	227.2	46.98	82	0.814	3.5	Nil.	Nil.
Combined Cuts[1] 10,11,12	0.883	460.4	59.01	82	0.814	7.0	0.044	Nil.

Note: (1) Oil heated with 4 wt. percent clay to $640^{o}F.$ over 60 minutes period with constant stirring in N_2 atmosphere. Cooled to $300^{o}F.$ in N_2 atmosphere and vacuum filtered. Clay was Pembina Clay R-105. Pembina Mountain Clays Corp. Winnipeg, Canada.

TABLE 6

Laboratory Distillation of Mohawk Waste Oil Treated as Shown in Table 2

(Lab Identification No. C-186)

Volume of Charge in Milliliters:	
Observed	3500 ml.
Calc. from wt. data	3500 ml.
Weight of Charge in Grams	3111 grams
Physical Inspections of Charge:	
Sp. Gr. 60°F./60°F.	0.889
Sulfated Residue, Wt.%	1.71
Coag. Pentane Insol., Wt.%	2.4
Weight Percent Solid NaOH to Charge	1.0

Cut No.	Pressure in inches of Hg. Vac.	Observed Liquid Temp. °F.	Observed[1] Vol. of Cut in Ml.	Measured Wt. of Cut in Grams.	Cumulative Percent Calc. from Wt. Data Vol.%[2]	Cumulative Percent Measured Wt.%
1	28.0	310-572	250	197.1	6.5	6.3
2	29.5	396-453	250	207.7	13.3	13.0
3	29.5	453-473	250	206.3	20.1	19.7
4	19.5	473-487	250	214.0	27.1	26.6
5	29.3	487-500	240	197.5	33.6	32.9
6	29.5	500-522	110	78.6	36.2	35.4
7	29.2	502-547	250	209.6	43.1	42.1
8	29.2	545-547	220	186.7	49.2	48.1
9	29.2	547-604	250	204.9	55.9	54.7
10	29.1	604-608	250	210.5	62.7	61.5
11	29.1	608-644	250	218.7	69.8	68.5
12	29.1	644-698	175	135.8	74.2	72.9
Bottoms	---	---	(455)	430.0	13.0	13.8
Loss					12.8	13.3

Note: (1) volumetric data are less accurate than weight data as it is difficult to withdraw sample from vacuum receiver without collecting additional sample after recording volume.

(2) calculated from weight data using gravities.

TABLE 7

Physical Inspections of Vac. Distillation Cuts from Table 6

(Lab Identification No. C-186)

Cut No.	Specific Gravity 60°/60°F.	Viscosity at 100°F. SUS	Viscosity at 210°F. SUS	Viscosity Index	Viscosity Gravity Constant	Color NPA Number	Sulfated Residue Wt.%	Coag. Pent. Insol. Wt.%
1	0.870	64.57	32.41	---	0.844	6.0		0.038
2	0.873	66.57	35.48	---	0.838	4.5		0.00
3	0.870	93.34	37.86	---	0.828	3.5		0.00
4	0.869	108.30	39.43	---	0.823	3.0	Nil	0.00
5	0.869	127.9	41.01	80	0.819	3.0	0.002	0.00
6	(0.870)	156.4	43.44	95	0.817	3.5	Nil	0.00
7	0.872	307.0(1)	42.34(1)	157	0.806(1)	7.0	0.002(1)	0.01(1)
8	0.881	318.5(1)	57.81(1)	122	0.818(1)	8.0+	0.46(1)	0.93(1)
9	0.876	242.0	47.11	71	0.816	3.5	0.036	0.00
10	0.878	309.8	52.60	90	0.815	3.5	Nil	0.00
11	0.879	401.2	57.43	93	0.811	4.0	0.003	0.00
12	0.882	490.7	62.70	96	0.812	6.0	0.01	0.00
Bottoms	0.947					Black	15.086	27.35

Note: (1) a slight upset in the distillation caused some bottoms to be carried over into these fractions.

TABLE 8

Physical Inspections of Clay Treated Vac. Distillation Cuts from Table 7

(Lab Identification No. C-186)

Cut No. (from Table 5)	Specific Gravity 60°/60°F.	Viscosity at 100°F. SUS	Viscosity at 210°F. SUS	Viscosity Index	Viscosity Gravity Constant	Color NPA Number	Sulfated Residue Wt.%	Coag. Pent. Insol. Wt.%
Combined Cuts[1] 4,5,6	0.866	121.6	39.98	52	0.816	1.5	Nil	Nil
Combined Cuts[1] 7,8	(0.874)	220.0	47.37	93	0.816	2.0	0.01	0.0012
Combined Cuts[1] 9,10	0.876	255.8	50.24	102	0.816	5.5	Nil	Nil
Combined Cuts[1] 11,12	0.880	443.5	59.63	93	0.811	8.0	Nil	Nil
Combined Cuts[2] 4,5,6	0.866	115.9	40.56	94	0.818	1.5	Nil	Nil
Combined Cuts[2] 7,8	0.874	217.7	46.95	90	0.816	0.5	Nil	Nil
Combined Cuts[2] 11,12	0.880	428.4	59.35	96	0.812	2.0	Nil	Nil

Note: (1) Oil heated with 2 wt. percent Fuller's earth to 640°F. over 60 minute period with constant stirring in N_2 atmosphere. Cooled to 300°F. in N_2 atmosphere and vacuum filtered. Clay was calcined Pikes Peak Clay No. 9475.

(2) Oil heated with 4 wt. percent filter clay to 640°F. over 60 minute period with constant stirring in N_2 atmosphere. Cooled over 30 minute period to 300°F. in N_2 atmosphere and vacuum filtered, Clay was Pembina Clay R-105.

TABLE 9

Laboratory Distillation of Mohawk Waste Oil Treated as Shown in Table 2

(Lab Identification No. S-1)

Volume of Charge in Milliliters:	
Observed	3500 ml.
Calc. from wt. data	3327 ml.
Weight of Charge in Grams	2951 grams
Physical Inspections of Charge:	
Sp. Gr. 60°F./60°F.	0.8871
Sulfated Residue, Wt.%	0.706
Coag. Pentane Insol., Wt.%	0.605
Weight Percent Solid NaOH Added to Charge	1.0

Cut No.	Pressure in inches of Hg. Vac.	Observed Vapor Temp. °F.	Observed Liquid Temp. °F.	Observed[1] Vol. of Cut in Ml.	Measured Wt. of Cut in Grams.	Cumulative Percent Calc. from Wt. Data Vol.%[2]	Cumulative Percent Measured Wt.%
1	27.9	293-450	329-446	132	128.5	4.5	4.4
2	26.8	450-529	446-572	250	189.5	11.0	10.8
3	26.8	529.572	572-608	250	198.0	17.8	17.5
4	27.8	572-586	608-631	250	201.0	24.7	24.3
5	28.2	586-595	631-648	250	206.0	31.6	31.3
6	28.4	595-610	648-671	250	207.5	38.9	38.3
7	28.4	610-635	671-703	250	209.0	46.0	45.4
8	28.4	635-662	703-720	250	205.0	53.0	52.4
9	28.4	662-693	720-752	175	144.0	57.8	57.3
Liquid in Trap				980	856.0	87.3	86.3
Bottoms					400	13.0	13.6
Loss						-0.3	+0.1

Note: (1) volumetric data are less accurate than weight data as it is difficult to withdraw sample from vacuum receiver without collecting additional sample after recording volume.

(2) calculated from weight data using gravities shown in Table 10.

TABLE 10

Physical Inspections of Vac. Distillation Cuts from Table 9

(Lab Identification No. S-1)

Cut No.	Specific Gravity 60°/60°F.	Viscosity at 100°F. SUS	Viscosity at 210°F. SUS	Viscosity Index	Viscosity Gravity Constant	Color NPA Number	Sulfated Residue Wt.%	Coag. Pent. Insol. Wt.%
1	0.866	35.42	17.70	---	---	4.5		Nil
2	0.871	76.63	36.35	---	0.833	4.5		Nil
3	0.872	114.4	39.88	---	0.827	4.5		Nil
4	0.876	148.6	42.56	86	0.824	5.0		Nil
5	0.877	189.1	44.34	72	0.821	5.0		Nil
6	0.[illegible]77	229.6	46.89	80	0.819	5.0		Nil
7	0.880	285.2	50.34	86	0.819	6.0	Nil	Nil
8	0.882	362.7	54.34	86	0.818	8.0	0.085	Nil
9	(0.898)	583.2	64.32	82	0.813	8.0+	--	--
Liquid in Trap	0.871	111.0	42.60	153	0.825		Nil	Nil
Bottoms	0.927							13.7

TABLE 11

Physical Inspections of Clay Treated Vac. Distillation Cuts from Table 10

Cut No. (from Table 8)	Specific Gravity 60°/60°F.	Viscosity at 100°F. SUS	Viscosity at 210°F. SUS	Viscosity Index	Viscosity Gravity Constant	Color NPA Number	Sulfated Residue Wt.%	Coag. Pent. Insol. Wt.%
Combined Cuts[1] 3,4,5	0.869	149.1	42.54	84	0.817	1.5	0.041	Nil
Combined Cuts[1] 6,7,8	0.877	248.0	49.94	104	0.818	3.0	0.0042	Nil
Cut No.[1] 9	0.896	717.5	70.74	84	0.826	8.0+	0.49	0.28

Note: (1) Oil heated with 6 wt. percent clay to 640°F. over 60 minute period with constant stirring in N_2 atmosphere. Cooled to 300°F. in N_2 atmosphere and vacuum filtered. Clay was Pembina Clay R-105.

TABLE 12

Conversion of Waste Oils into Heating Oils

Raw Waste Oil Sample No.	Origin of Waste Oil	Dehydration Temperature, °F.
Sample 11	Drum from Mohawk Oils Refining Co., Newark, New Jersey, received 6/1/67	350°
Sample 10	Drum from Riegel Petroleum Co., Bridgeport, New Jersey, received 5/19/67	400°
Sample 7	Gallon Sample from Villanova Citgo Truck Service Station collected 4/27/67	350°
Sample 6	Gallon Sample from Sunoco Service Station collected 4/25/67	350°
Sample 8	Five Gallon Sample from Tidewater Flying A Station collected 5/4/67	350°
Sample 9	Drum from Berks Associates, Douglasville, Pa., received 5/9/67	400°

TABLE 12 (continued)

Conversion of Waste Oils into Heating Oils

Raw Waste Oil Sample No.	Additional Treatment of Dehydrated Oil	Settling Time Before Sharples Centrifugation Hours	Sharples[1] Centrifuge Speed	Solvent Stripping Temp. °F.
Sample 11	3:1 Udex Raffinate to Oil at 150°F. Mixed 15 min. Settled 1.5 hours. Put thru CRC Auto Filter[2]	1.0	6,700 G	400
Sample 10	4:1 Udex Raffinate to Oil at 150°F. Mixed 15 min. Settled 1.0 hour. Put thru CRC Auto Filter. Then 4.5 wt.% Atlas Filter Carbon S-1[3] added and again put thru Auto Filter[2]	1.0	6,700 G	400
Sample 7	4:1 Udex Raffinate to Oil at 150°F. Mixed 15 min. Settled 1.0 hour.	1.0	6,700 G	400
Sample 6	3:1 Udex Raffinate to Oil at 150°F. Mixed 15 min. Settled 1.0 hour.	1.0	6,700 G	400
Sample 8	3:1 Udex Raffinate to Oil at 150°F. Mixed 15 min. Settled 1.0 hour.	1.0	6,700 G	400
Sample 9	3:1 Udex Raffinate to Oil at 150°F. Mixed 15 min. Settled 1.0 hour.	1.0	6,700 G	400

Note: (1) Sharples laboratory model super-decanter centrifuge
(2) Laboratory open bowl filter spun to drive liquid thru filter paper at ca. 700 G.
(3) Grade S-51 Darco filtering carbon made by Atlas Chemical Corp.

TABLE 13

Physical Inspections of Waste Oils Shown in Table 12

Raw Waste Oil Sample No.	Physical Inspections of Raw Oils:	Physical Inspections of Dehydrated Oils:	Coagulated Pentane Insol. of Additionally Treated Oils:	
	Water by Distillation, Vol.%	Coagulated Pentane Insolubles, Wt.%	After CRC Filter and Solvent Stripping, Wt.%	After Sharples Centrifuge and Solvent Stripping, Wt.%
Sample 11	11.0	3.3	After CRC Filter with Filtering Clay Pre-coat = 1.5	0.07 Duplicate Sample 0.13
Sample 10	2.2	1.7	After 1st CRC Filter = 1.7 After 2nd CRC Filter = 1.7	
Sample 7	0.1	7.8		
Sample 6	Nil	1.9		
Sample 8	Nil	6.2		
Sample 9	Nil	3.3		

TABLE 14

Physical Inspections of Sharples Centrifuged and Solvent Stripped Oils Shown in Table 12(1)

Raw Waste Oil Sample No.	Gravity °API	Viscosity at 100°F. SUS	Viscosity at 210°F. SUS	Viscosity Index	Viscosity Gravity Constant	Flash, O.C., °F.	Fire, °F.	Pour, °F.	Sulfated Residue, Wt.%	Ramsbottom Carbon Residue, Wt.%
Sample 11	28.7	209.5	50.08	128	0.818	325	345	-25	0.66	1.44
Duplicate Sample 11	28.7	205.8	49.92	129	0.819	340	385	-30	0.68	1.44
Sample 10	29.6	275.5	53.93	119	0.810	445	470	-25	0.49	1.10
Sample 7	27.4	285.2	54.51	118	0.825	335	385	-35	0.95	1.93
Sample 6	28.6	200.3	50.85	138	0.819	300	330	-30	0.91	1.81
Sample 8	27.7	273.2	54.41	123	0.822	380	410	-30	0.84	1.86
Sample 9	28.4	207.7	49.85	128	0.822	315	405	-30	0.56	1.32

Note: (1) Samples were submitted to Mr. W. K. Griessinger, Director of Research and Development of Atlantic Refining Co. and test data was obtained under the supervision of Mr. L. Way of the Research and Development Division on Sept. 21, 1967.

TABLE 15

Physical Inspections of Heating Oils Blended from Stocks of Table 14[1]

Raw Waste Oil Sample No.	Cutter Stock[2] Added Vol.% of Blend	Physical Inspections of Blends with Cutter to Meet No. 4 Heating Oil Viscosity Specifications[2]		
		Viscosity at 100°F. SUS	SUS at 100°F. Required for F.O. No. 4	Sulfur, Wt.% ASTM-D-1551-65T
Sample 11	23.0	99.2	45 to 125	0.34
Duplicate Sample 11	22.0	103.0	"	0.32
Sample 7	28.5	99.6	"	0.39
Sample 6	22.0	100.6	"	0.37
Sample 8	28.0	100.3	"	0.38
Sample 9	23.0	98.4	"	0.26

Note: (1) Samples were submitted to the Gulf Oil Corporation, Philadelphia Refinery, and test data was received through the courtesy of Mr. D. R. Clarke, Chief Chemist, on Nov. 10, 1967.

(2) Cutter was standard fuel blending stock having the following physical inspections:

Viscosity, SUS at 100°F.	35	
Sulfur, Wt.%	0.10	
Sulfated Residue, Wt.%	0.001	Max.
Ramsbottom Carbon Residue, Wt.%	0.016	
Neutralization Number	0.01	
Flash, O.C., °F.	160	Min.
BS and W, Vol.%	0.01	
Gravity, °API	33.0	

APPENDIX

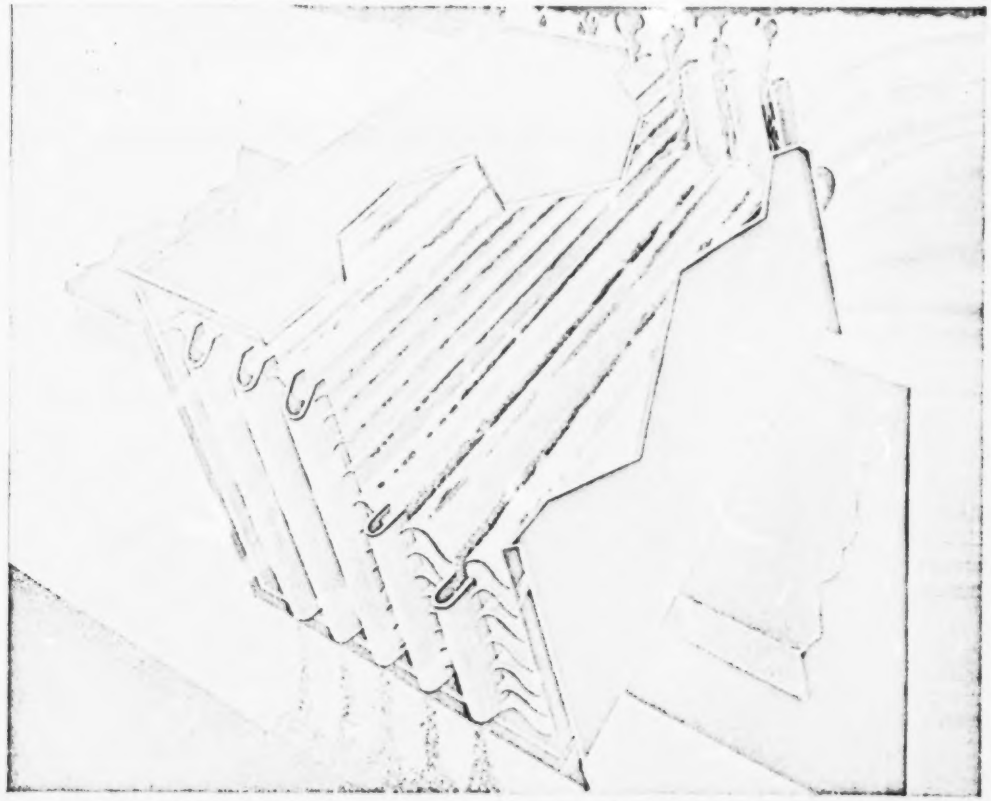
Manufacturing Specifications for Clay Treated Lube Oil Stocks of Berks Associates, Inc.

Vacuum Tower Cut	Viscosity at 100°F SUS	Viscosity at 210°F SUS	Viscosity Index	Color NPA Number
Cut 1 Clay Treated	Min. 80	Min. 36	Min. 80	Min. 2.0
	Max. 100	Max. 40	Max. 100	Max. 3.5
Cut 2 Clay Treated	Min. 100	Min. 40	Min. 80	Min. 2.5
	Max. 200	Max. 47	Max. 100	Max. 3.5
Cut 3 Clay Treated	Min. 200	Min. 48	Min. 80	Min. 3.0
	Max. 500	Max. 58	Max. 100	Max. 6.0

TPS

TILTED PLATE SEPARATOR

- Het platenpakket kan onder een steilere hoek worden gesteld, zodat het sediment beter afglijdt.
- De plaatafstand kan naderhand vergroot worden.



FIGUUR C

TOELICHTING OP DE FIGUREN

Het TPS kan worden toegepast voor de afscheiding van deeltjes lichter dan de continue fase. Het proces geschiedt dan volgens fig. A, dan wel van deeltjes zwaarder dan de continue fase (fig. B). De werking van bestaande afscheiders kan veelal aanzienlijk worden verbeterd door bijplaatsing van een TPS. (zie als voorbeeld fig. C)

BERKS ASSOCIATES, INC.

PETROLEUM CONSERVATIONISTS

DOUGLASSVILLE, PA.
PHONE - 385-3031

P. O. BOX 617
POTTSTOWN, PA.
19464

January 6, 1968

Dr. Riley N. Kinman, Assistant Chief
Demonstrations Grants Branch
Division of Research and Training Grants
Federal Water Pollution Control Administration
633 Indiana Avenue N.W., Room 916
Washington, D. C.

Dear Dr. Kinman:

We are interested in Dr. E. W. Clarke's method of rerefining waste oil to eliminate the dangers of polution of streams and waterways. The work he has shown us makes us feel that he has a worthwhile procedure.

During the past several years we have been rerefining waste oil without the use of sulfuric acid, through the use of a vacuum still but have not found a satisfactory method of utilizing the waste materials, consisting of additives and other carbon deposits. We are convinced that the waste material is as valuable as the oil itself, not to mention the elimination of the pollutants. Dr. Clarke has given us an assurance that with a further study of this problem he will find a satisfactory and economical use for the bottoms.

We are presently making alternations to our plant to put through a plant run to verify Dr. Clarke's laboratory findings and hope to have the first run on or about January 16, 1968. If this is of interest to you call or write to me at the above address for our experience.

Very truly yours,

BERKS ASSOCIATES, INC.

Roy H. Schurr

Roy H. Schurr, President

cc: Dr. E. W. Clarke
121 Fairmount Avenue
Laurel Springs, N. J. 08021

BERKS ASSOCIATES, INC.

PETROLEUM CONSERVATIONISTS

DOUGLASSVILLE, PA.
PHONE - 385-3031

P. O. BOX 617
POTTSTOWN, PA.
19464

March 9, 1968

Capt. Henry T. Koonce, M.S.
Director of Research and Development
Villanova University
Villanova, Pennsylvania

Dear Capt. Koonce:

Confirming my conversation with you on March 8, 1968, I wish to place on record a report that we are now producing a satisfactory grade of lubricating oil, made from crankcase oils, with practically no contamination of the effluent or the air. Presently we are charging 25 gallons per minute of dehydrated oil to our stills and on March 7, 1968 we produced 19,000 gallons of finished lubricating oil having a No. 2 color. We are still in the process of installing larger and more complete equipment and feel one of our next problems will be to secure sufficient crankcase oil to keep our plant in operation. We are using but one chemical which is not injurious or harmful to the waters, in fact our PH will come out neutral, and I believe with our present results we will have something which will be very pleasing and satisfactory.

For your information we have been re-refining crankcase oil without the use of sulfuric acid for over four years but our equipment has not always been capable of producing our entire requirement. Therefore, we are occasionally forced to use sulfuric acid to fill some of our orders. We are quite sure though that this doesn't always produce a good re-refined oil.

We can only say that until we had the pleasure of meeting Dr. Edward Clarke we were giving serious consideration to discontinuing the business. I am happy to report that Dr. Clarke has been a new inspiration to me and I am now planning on making considerable changes to our plant to complete his work. May I thank you for Dr. Clarke's services if this will pay the bill.

Thanking you for the pleasure and cooperation shown during our telephone conversation and I want you to feel that you are personally welcome anytime at our plant in the accompaniment of Dr. Clarke.

Very truly yours,

BERKS ASSOCIATES, INC.

Roy H. Schurr

Roy H. Schurr, President

RHS/jy

cc: Dr. E. W. Clarke
121 Fairmount Avenue
Laurel Springs, N. J.

April 26, 1968

Capt. Henry T. Koonce, M.S.
Director of Research and Development
Villanova University
Villanova, Pennsylvania

Dear Captain Koonce:

As a further report on my letter of March 9, 1968, the plant run of the used crankcase oil is showing a satisfactory result and all indications are favorable to an increase in earnings. Much work and plant adjustment will increase the earnings to a satisfactory basis that will be more profitable than at any time we used sulfuric acid, not to mention the elimination of water and air pollution and plant deterioration. Your Dr. E. W. Clarke has been our constant source of help in rearranging the routing of the flow of the oil in process and feel absolute confidence in his suggestions.

Presently we are dehydrating with the same equipment we used in the sulfuric acid treatment but we now use a by-product of our process to dilute the dirty crankcase oil and add some common chemicals to start the dissolution of the undesirable materials. After this it goes through equipment that we have developed to further the dissolution of the heavy ends. It is then atmospherically distilled to remove the by-product we used to create the dilution. From this still it is vacuum distilled to recover the lubricating oils that will be clay treated to complete desired discoloration of the oils.

Also, there is still some additional equipment to be installed to completely recover the additives and carbon residues. It will require several months to complete the entire rebuilding but feel that there is sufficient results to warrant continuing the development of a complete plant running on automation.

We have already mailed you a comparative run sheet to show you some of the results obtained to date.

You are welcome to visit our plant at anytime, however, I would appreciate if you would call me prior to your arrival so that I will be on hand to meet you.

Very truly yours,

BERKS ASSOCIATES, INC.

Roy H. Schurr

Roy H. Schurr, President

RHS/jy

cc: Dr. E. W. Clarke
121 Fairmount Avenue
Laurel Springs, New Jersey

April 26, 1968

Capt. Henry T. Koonce, M.S.
Director of Research and Development
Villanova University
Villanova, Pennsylvania

Dear Captain Koonce:

Following are some tabulations of production runs showing the difference in clay cost based on our delivered cost of $1.90 per 50 lb. bag. We also are no longer using sulfuric acid that was costing at least $2,000.00 per month plus disposing of the bottoms. Our chemicals now being used will cost about $800.00 per month.

This tabulation represents the daily oil produced and the clay used in the month of March.

Oil Produced	Clay
15,800	182
3,350	28
8,530	149
19,000	149
16,930	153
12,870	142
16,700	135
20,350	147
12,150	159
16,800	171
16,300	164
20,800	125
15,600	123
1,050	14
8,400	113
12,600	195
16,800	175
13,120	209
11,460	135
6,550	161

This tabulation represents the daily oil produced and the clay used in the first 14 days of April.

Oil Produced		Clay
12,000		120
27,450		227
27,200		224
25,350		207
21,900		174
1,450		11
13,500		159
24,000		210
20,000		171
25,050		204
21,500		173
27,400		152
15,650		115
10,750		100
273,200	TOTALS	2,247

(continued on page 2)

8,720		193
11,900		201
8,670		201
9,280		124
23,250		244
19,550		194
2[illegible],500		193
18,400		228
388,430	TOTALS	4,407

In computing the clay savings we find we produced 88 gallons of oil for every bag of clay used in March 68.
In April we produced 121 gallons of oil for every bag of clay with a slightly better color.

Trusting this will indicate to you the advantages of producing an oil without the use of sulfuric acid.

Very truly yours,

BERKS ASSOCIATES, INC.

R H Schurr

Roy H. Schurr, President

RHS/jy

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SCHOFIELD & deVRIES LIMITED

BRESLAU · ONTARIO

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BRESLAU, ONTARIO
Box 15
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Direct Toronto Line
416-363-9197

TORONTO, ONTARIO
115 Unwin Ave.
416-461-6354

WINNIPEG, MANITOBA
Lubrifine Division
Mission St. & 59 Hwy.
204-247-2646

Cable Address
SKOIL

April 30, 1968

Dr. Edgar W. Clarke
121 Fairmount Avenue
Laurel Spring, New Jersey 08021

Dear Dr. Clarke:

I have been extremely interested in your work at Villanova University regarding your new process for the refining of Automotive Crankcase Drainings without the use of Sulphuric Acid. Equally important is your work on the recovery of additives and the charring of sedement wastes, turning them into a saleable item instead of one more factor to the Pollution Area.

I am pleased to report that on April 3, 1968, our Toronto Plant initiated phase one of your process: that of Dehydration (250-300°F.) and Caustic Treating. This new step resulted in the saving of 40% on our acid consumption. This is most significant from cost savings standpoint, but more important, it has greatly reduced our acid sludge disposal problem.

I am looking forward to implementing phase two and three and the ultimate elimination of acid altogether; to say nothing of the added revenue to be derived from additive recovery and charr by-products.

Your work at Villanova has been most gratifying and you must surely share a feeling of great accomplishment. I know our industry and the nation as a whole will benefit greatly at this time of great pollution peril.

It is with a great deal of pleasure I have written this letter to express our appreciation for your most sincere interest in our problem and your unselfish cooperation with us when we would call you. We wish you every success in your future endeavors.

Best personal regards

Robert F. Thomas

R.F. Thomas
PLANT MANAGER

RFT/dc

PETROLEUM PRODUCTS

The story of... RE-REFINED OIL

...As produced by

MOTOR OILS REFINING COMPANY

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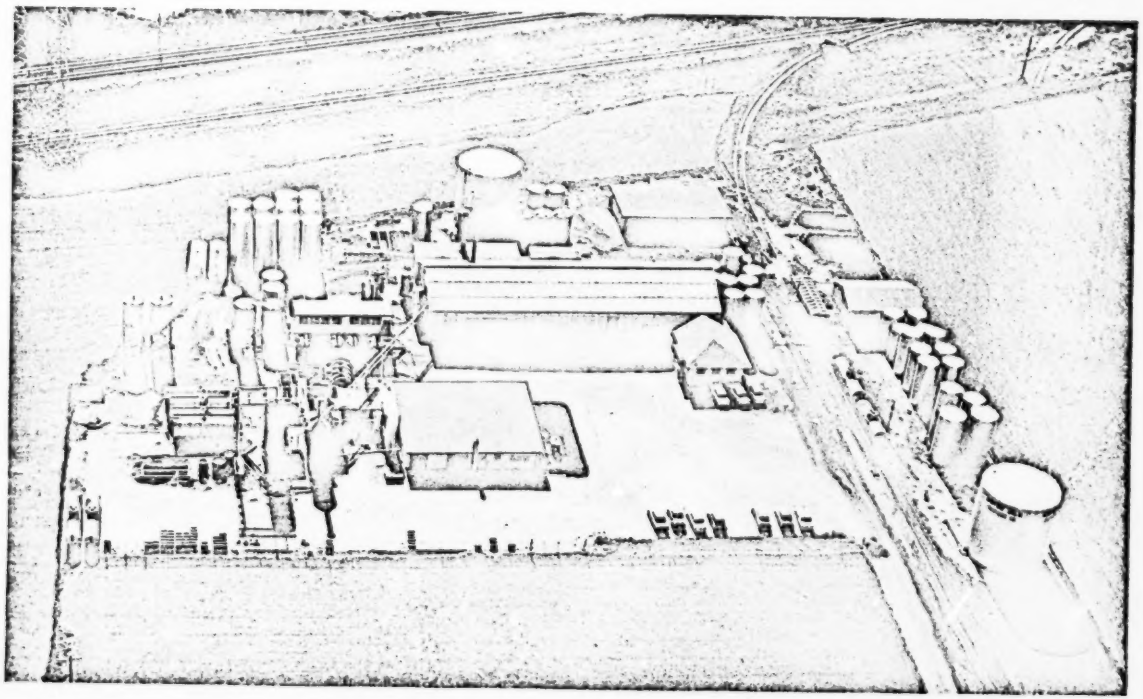
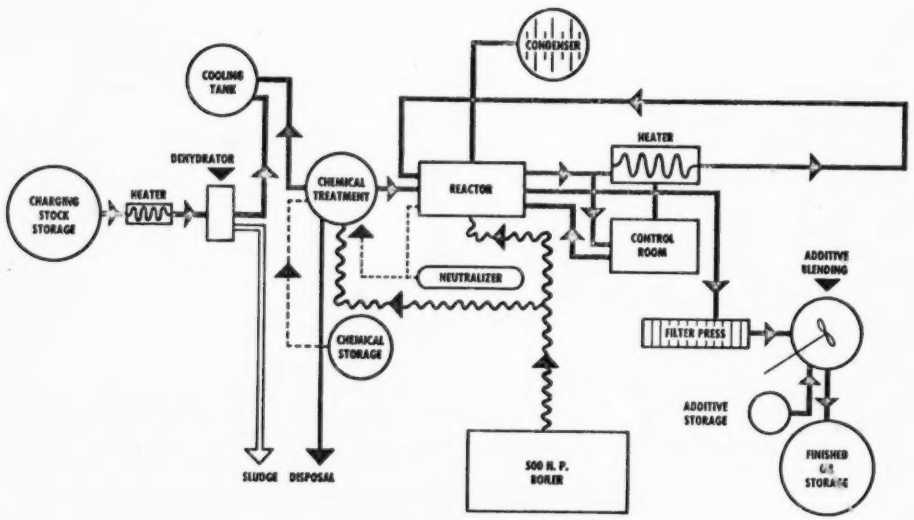
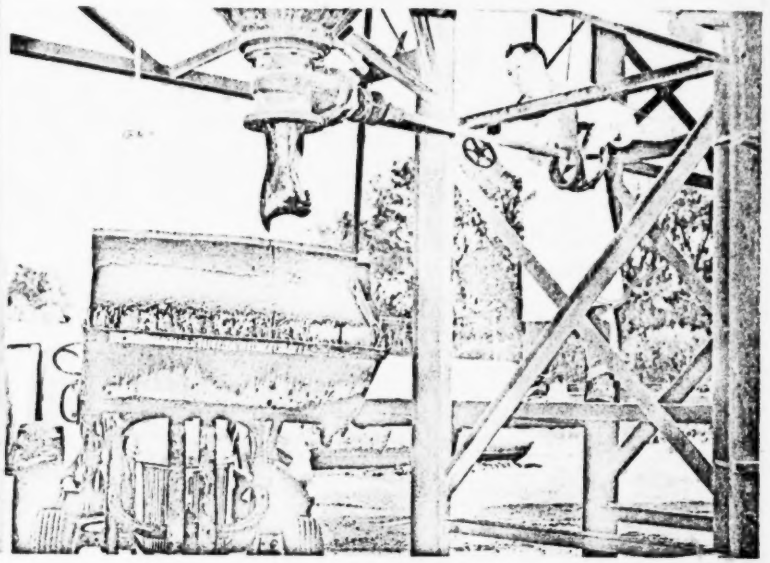


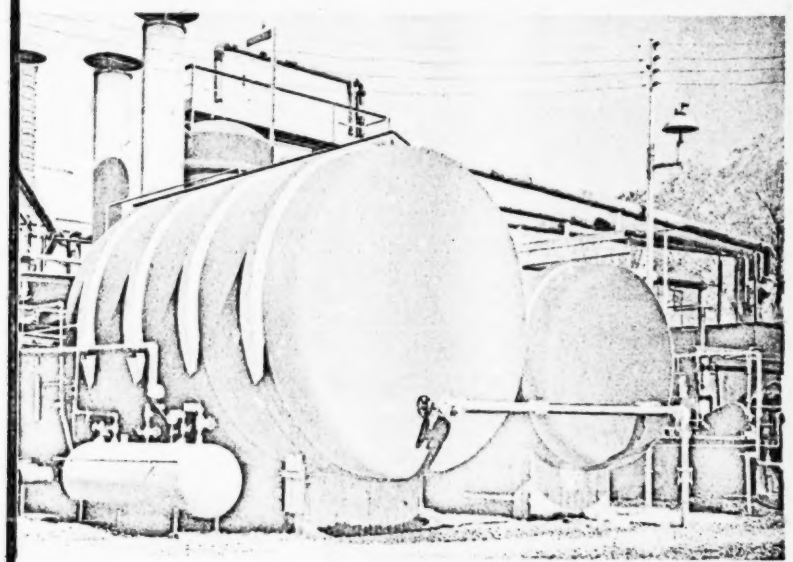
. . . how it's done

What is re-refined oil? — When oil is used it becomes adulterated with extraneous solid and liquid materials that include products of oxidation, combustion and dirt. The re-refined oil produced by our firm is oil that has had these foreign contaminants removed in their entirety by our thorough re-refining process. This process imparts superior oxidation stability to the oil which, in many instances, exceeds that of the virgin oil. This is due to the fact that the less stable hydrocarbon components that have oxidized in use, are removed during re-refining to yield a most stable lubricant. Depicted below is a flow diagram for re-refining oil.

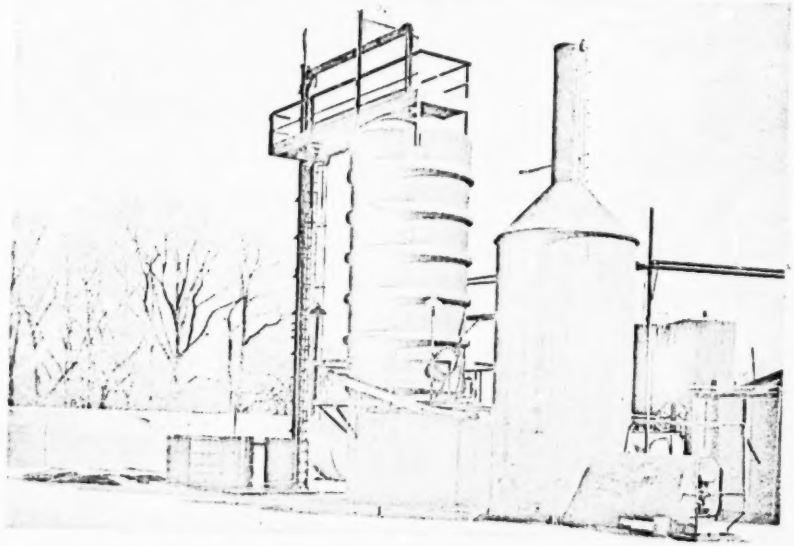


CHARGING STOCK
CHEMICALS
WATER
STEAM
SLUDGE





The used oil which we re-refine is first thoroughly dehydrated. This prepares it for de-sludging which is accomplished by chemical treatment. We remove sludge, oxidized materials, resins and other undesirable materials in this phase of our process. Can you imagine the harmful effects resulting from circulating sludge such as this through your engine?



A larger reactor of 10,000 gallons capacity is shown at left.

During this heating cycle, all low boiling fractions such as gasoline, fuel oil and other diluents, which are present in the used oil, are distilled off in the form of vapors as their respective boiling points are reached in the reactors.

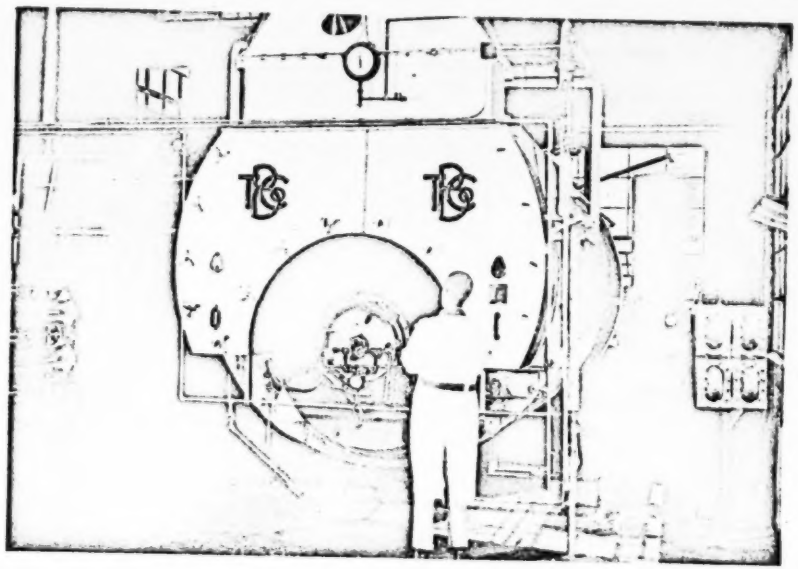
REDUCE YOUR COSTS -- USE RE-REFINED OIL

4e

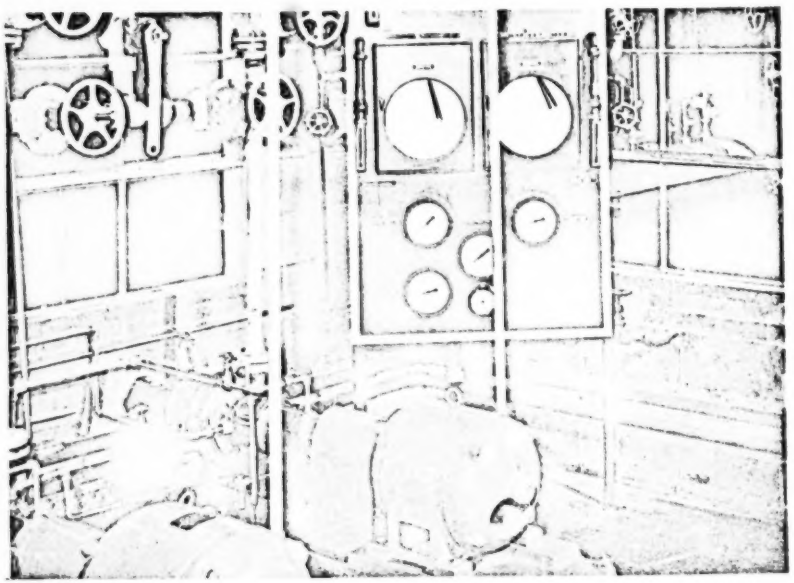
Following the removal of sludge the oil is pumped to the reactors of our distillation units.

Shown at left are our two reactors with capacities of 4500 and 9000 gallons. Each reactor is equipped with a heating unit of sufficient capacity to heat the oil to the required temperature.

Reproduced from best available copy.



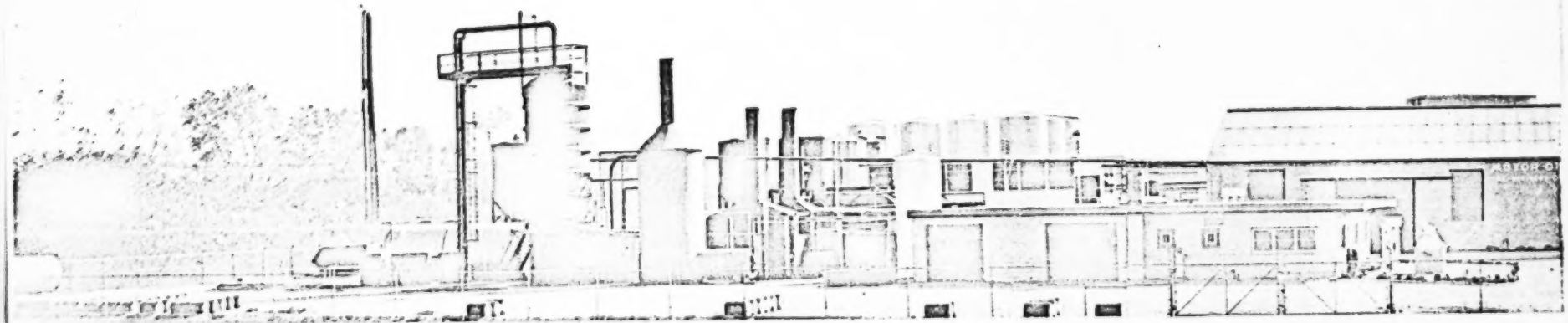
Steam is used very generously in the re-refining process to help remove the low boiling fractions, together with other undesirable elements. Since an abundance of steam is required, it is amply supplied by a 500 Horse Power capacity boiler shown here.



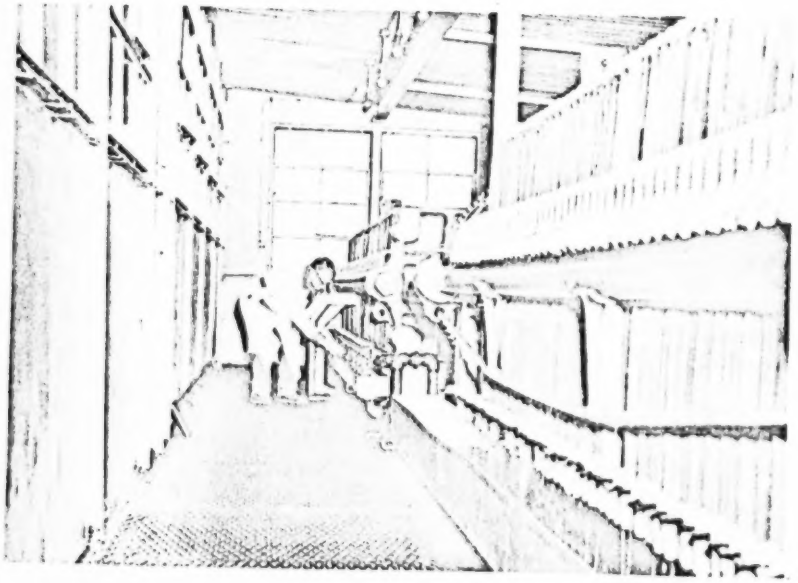
During the distillation cycle, in which the oil in the reactors is heated and distilled, a clarifying agent is injected into the stream in the prescribed amount. The function of this material is to adsorb all remaining extraneous contaminants contained in the oil which is a step in restoring the purity and color of the oil.

Our control room is equipped with the necessary pumps, recording instruments, gauges, valves, etc., to enable us to properly regulate the oil flow, the temperature, amount of steam injected, etc., during distillation. When our controlling specifications are attained the oil is cooled and pumped to our filter presses which consist of two groups, primary and secondary.

47

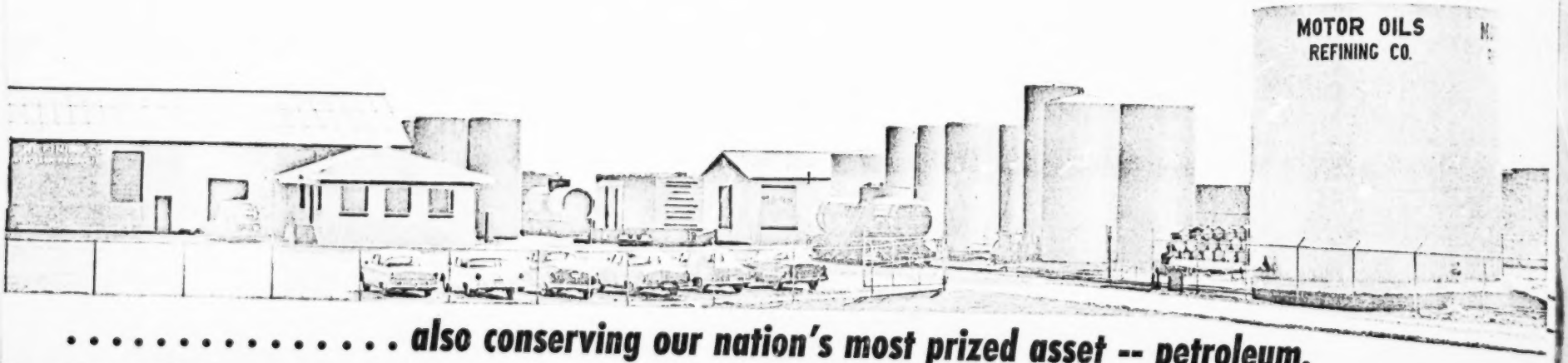


Expanding to supply an increasing demand for high quality products at economical prices . . .



Here all impurities are completely removed. We employ the use of a secondary filter to assure us of thorough filtration and to further polish the finished oil.

The additives such as detergents, dispersants, and oxidation inhibitors, are accurately metered into the re-refined oil and both are thoroughly mixed in the blending tanks. Special type of additives are available should a customer need it for certain requirements.



. . . . . also conserving our nation's most prized asset -- petroleum.

DEPARTMENT of the INTERIOR

news release

OFFICE OF THE SECRETARY

Daitch--962-8938

For Release to PM's March 15, 1968

INTERIOR SUBMITS SUGGESTED LEGISLATION TO CONTROL OIL POLLUTION

The Department of the Interior has submitted to Congress proposed legislation to help control pollution of the Nation's waters by oil spills and other hazardous substances, Secretary of the Interior Stewart L. Udall announced today.

The President proposed the enactment of such legislation in his recent message to Congress, "To Renew A Nation."

The measure would carry out some of the major recommendations made in a detailed report, "Oil Pollution--A Report to the President," which was prepared jointly by the Department of the Interior and Department of Transportation at the request of the President. Other recommendations made in the report are already included in S. 2760 which the Senate passed in December 1967.

The main provisions of the proposed legislation are as follows:

--Prohibits the discharge of oil if it occurs from a shore facility or a ship operating within 12 miles from shore. The 3-mile territorial and 9-mile contiguous zones are thus both covered. This greatly expands the previous standard of liability, which was limited to "gross or willful negligence" and to the 3-mile limit.

--Imposes upon the discharger of oil including ship and shore facility owners the responsibility for cleaning up the affected areas.

--Empowers the Federal Government to clean-up oil spills whenever the owner or operator fails to act, but requires the pollutor to reimburse the Government for the clean-up costs. Prior law limited the owner's liability to the salvage value of the ship. The proposal will make them liable for the full costs of clean-up.

--Provides protection against large and dangerous discharges of pollutants other than oil by requiring those responsible to take whatever clean-up or other action the Government considers necessary. If the pollutor fails to act, the Government will take the necessary steps, and hold the pollutor liable for the costs.

The report by the Department of the Interior and Department of Transportation pointed out to the President that the risks of oil contamination and other hazardous substances are as numerous and varied as the uses made of them. The report also said that wherever oil is used, stored, or moved, it is subject to mechanical failures compounded by human carelessness and mistakes.

Referring to the Torrey Canyon disaster, the report said: "Because sizable spills are not uncommon, and major spills are an ever-present danger, effective steps must be taken to reduce this Nation's vulnerability."

# # #

3165-68

Laboratory Study of Vacuum Tower Bottoms

Experiment A

A 254 gram sample of vacuum residue from the distillation shown in Table 6 was stirred at 190°F. with 1200 ml. of Udex Raffinate and filtered. The solid residue, after drying to constant weight, was 9.26 weight percent of the vacuum residue. The filtrate was heated to 400°F. to strip off the Udex Raffinate. The stripped oil was treated with 4 weight percent of Pembina Clay R-105 at 640°F. and then with 8 weight percent of Grade S-51 Darco filtering carbon (made by Atlas Chemical Corp.), and again filtered. The results are as follows:

	Vacuum Bottoms from Table 6	Udex Treated and Clay Treated Vacuum Bottoms
Sulfated Residue, Wt. %	15.1	11.0
Coagulated Pentane Insolubles, Wt. %	27.4	14.1

From the above data, it was concluded that the sulfated residue was not reduced to an acceptable level for burning as an industrial fuel. Neither precipitation of solids from Udex Raffinate solution, nor clay treatment, nor filtration with filtering carbon had been effective. Since the vacuum tower bottoms produced commercially at Berks Associates, Inc. after neutralization, dilution with light oil, and filtration, is an acceptable plant fuel, it appears that neutralization of the vacuum residue before dilution and filtration is an absolute necessity in order to produce a fuel of acceptable quality for plant use.

Experiment B

A 172 gram sample of vacuum residue from the distillation shown in Table 9 was stirred at 134°F. with 2300 ml. of acetone and filtered. After heating the filtrate to strip off the acetone, a 29 weight percent yield of a golden-red liquid was obtained (6 NPA color). The sulfated ash was 0.4% and the coagulated pentane insolubles was 0.66%. The viscosity at 210°F. in SUS was 133.6. The high value of viscosity along with the low values of sulfated residue and coagulated pentane insolubles suggests that the valuable detergent - V.I. improvers in the used oil have been concentrated in this fraction. The commercial separation and re-use of this material is an exciting possibility for the re-refiner.